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I, ANNA MAIJA MADL, ACTING TEAM LEADER EXAMINATION SUPPORT & SALES hereby certify that annexed is a true copy of the Provisional specification in connection with Application No. PP 6025 for a patent by M.I.M. HOLDINGS LIMITED filed on 21 September 1998.

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AUSTRALIA  
*Patents Act 1990*

**PROVISIONAL SPECIFICATION**

**Invention Title: METHOD FOR TREATING PRECIOUS METAL BEARING  
SULPHIDES**

**This invention is described in the following statement:**

## TITLE

## METHOD FOR TREATING PRECIOUS METAL BEARING SULPHIDES

## FIELD OF THE INVENTION

The present invention relates to a process for  
5 leaching refractory sulphide ores or concentrates and in  
particular is directed towards a method of recovering  
precious metals contained in the ores or concentrates.

## BACKGROUND ART

Gold is generally extracted from gold  
10 containing ores by treatment with a cyanide solution  
which solubilizes the gold. However, in some ores, the  
gold is present as microparticles encapsulated within the  
ore. The gold in such ores cannot be extracted by  
traditional cyanidation techniques. These types of ores  
15 are known as refractory ores and are typically sulphide  
ores.

In order to extract gold from refractory ores,  
the ores must first be treated to liberate the gold so as  
to be accessible to cyanide leaching. A refractory ore  
20 is typically treated by oxidizing the ore which results  
in the chemical destruction of the sulphide matrix. Known  
methods of oxidising refractory ores include leaching the  
ore at elevated temperatures and pressure under acidic  
conditions. An example of such a process is known as the  
25 Sherritt process which includes the steps of feed  
preparation, pressure oxidation in the presence of  
oxygen, solid/liquid separation, liquid neutralization  
and gold recovery from the oxidised solids by  
cyanidation. The operating conditions required in this  
30 process are at temperatures of between about 150°C to  
210°C, a total pressure of 2,100 kPa, a pulp density of  
20% to 30% solids by mass and a retention time of two to  
three hours. The oxidation must be carried out in  
autoclaves and requires a source of oxygen. The capital  
35 costs associated with providing the autoclaves meeting a  
high oxygen demand are high and may be prohibitive for  
construction at remote sites, for medium to small scale  
operations and for low grade ore. It is possible to

leach under less aggressive conditions but in this case, the leaching rates and recovery are too low to be economically viable.

Attempts have been made to reduce the aggressive conditions and to lower the pressures and temperatures required whilst maintaining the economic viability of the process. For example, Australian patent application no. 27182/92 describes subjecting an ore concentrate to fine grinding prior to leaching. Fine grinding to an 80% passing size of 15 micron or less enabled the leaching to be carried out under less aggressive conditions at temperatures of 95-110°C and pressures of about 1000 kPa.

Thus, whilst some progress has been made in reducing the operating parameters when using oxygen as the oxidant, the leach must still be carried out under pressure.

It is also known to oxidatively leach mineral species with ferric ions under acidic conditions. Ferric ion is typically a more effective oxidizing agent than oxygen which means that oxidation with ferric ions can be carried out under less aggressive conditions. Oxidative leaching at atmospheric pressure using ferric ions is known. A disadvantage of leaching with ferric ions is that the ferric ions are reduced to ferrous ions during the leaching reaction. An increase in ferrous ion concentration adversely affects the rate of leaching. Thus, it is necessary to remove the ferrous ion during the leach. Further, as leaching solutions are recycled, ferric ions must be regenerated by oxidizing the reduced ferrous.

A common disadvantage with the aforementioned processes is that they operate under acidic conditions. As cyanidation requires alkaline conditions, the acid must be removed prior to cyanidation. This may be overcome by leaching under alkaline conditions and such leaching is known. However, a major disadvantage of alkaline leaching is that when iron containing ores such

as pyrite are leached, the sulphide iron which is leached precipitates as a passive iron oxide layer on the mineral particle. This layer inhibits further oxidation with the result being that the extent of leaching under alkaline conditions is less than under acidic conditions. This translates to a lower recovery of precious metals.

Still further, alkaline leaching also requires elevated pressure and temperatures and an oxygen source for the leaching to occur at a rate and provide gold recoveries which may be economically viable. However, even under aggressive conditions, recovery of precious metals is less than that for acid leaching.

Further, base metals such as copper and zinc are insoluble at high pH. Thus, alkaline leaching is unsuitable for leaching ores or concentrates where recovery of base metals from base metal sulphides such as chalcocite, sphalerite or chalcopyrite is required. For these reasons, commercial and academic interest has been directed towards acid leaching.

Most of the literature relating to alkaline leaching is directed towards the use of alkalis such as sodium or potassium hydroxide and ammonia. A disadvantage with these reagents is that iron is precipitated primarily as jarosite. Jarosite inhibits gold recovery and is also an environmentally unacceptable residue. Also, hydroxide reagents and in particular sodium hydroxide are prohibitively expensive.

The use of cheaper alkalis such as lime has been proposed. However, to date, leaching of iron sulphide materials with lime has been unsuccessful in that leaching is incomplete and subsequent precious metal recovery is low. For example, an earlier study of alkaline oxidation of pyrite in gold recovery using lime achieved only 30 to 40% gold recovery which offered little improvement over direct cyanidation of the pyrite. This is believed to be due to passivation of the mineral by precipitation of an alkali/gypsum/iron oxide layer. Limestone is another alkali which is relatively cheap.

Limestone is typically used in the neutralization of acidic leachates. However, limestone is considered to be insufficiently reactive and/or soluble in alkaline systems to be able to be used for alkaline leaching.

5           From an economic point of view, it would be desirable to be able to leach refractory materials for precious metal recovery under mild alkaline conditions and using reagents other than the expensive hydroxides. As mentioned above, it is known that the oxidation rate  
10 under acidic conditions can be increased by fine grinding to increase the surface area of the mineral particles. Such an increase may be predicted given that there is a larger surface area exposed to the oxidizing agents. However in the alkaline system, this is not the case in  
15 view of the formation of the passive iron oxide layer which forms on the surface of particles. The rate determining factors in the alkaline systems rates are believed to relate to the formation of the passivating iron oxide layer and diffusion of oxygen through the  
20 layer. Thus, workers in the field have concentrated on increasing the extent of alkaline leaching by modifying the leaching conditions so as to minimise formation of the passive layer and/or influence the diffusion rate through the layer. One study suggests leaching at higher  
25 temperatures or at relatively concentrated solutions of reagents. The reason for this is to rapidly produce a passive layer which is unstable and subject to cracking.

It is believed that at lower temperatures, the layers grow more slowly and are more stable. Another suggestion  
30 has been to use additives which may react to dissolve the layer or to make the layer more permeable.

However, to date no method proposed has been proposed which is able to successfully leach iron containing refractory ores under alkaline conditions  
35 which also enables good recovery of precious metals from the ore.

The present invention is based on the surprising and unexpected discovery that leaching an iron



containing refractory material under alkaline conditions can be successfully achieved by careful selection of the particle size of the material to be leached. Even more surprising it has been discovered that not only can the  
5 leaching efficiency be improved but that leaching can also be successfully conducted under relatively mild conditions.

According to a first broad form of the invention there is provided a method of processing a  
10 mineral composition comprising an iron containing refractory sulphide, the method comprising milling the composition to a particle size of  $P_{80}$  of at least 25 microns and leaching said composition with a solution comprising lime and/or limestone in the presence of an  
15 oxygen containing gas.

The method of the present invention is applicable to a composition including an iron containing refractory sulphide material or a mixture of such materials. Examples of such materials include pyrite,  
20 arsenopyrite and pyrrhotite. Other non-iron materials may also be present in the composition, examples of which include stibnite, tetrahedrite, tennantite and pyroargenite. Suitably the composition would not include economic amounts of base metal sulphides containing  
25 copper or zinc. Generally a composition which includes appreciable amounts of these base metals would not be treated by the method of the present invention. The reason for this is purely economical as copper and zinc precipitate under alkaline conditions and thus cannot be  
30 recovered easily. Under acidic leaching conditions, copper and zinc are solubilized and can be recovered by conventional SX/EW techniques.

The method of the present invention is in particular directed towards the treatment of refractory  
35 materials containing precious metals such as gold, silver and platinum. Suitably, the refractory materials are in the form of ores flotation concentrates.

In the method of the present invention, the

composition is finely ground to a particle size of  $P_{80}$  at least 25 $\mu$ m. A typical particle size range is between 5-25 $\mu$ m and preferably between about 12-20 $\mu$ m.

5 A preferred apparatus for producing the finely ground material is a stirred mill. However, it will be appreciated that other types of comminution apparatus may also be used such as wet and dry vibratory mills or planetary mills.

10 The leaching is carried out using lime and/or limestone as the alkali reagent. Lime, limestone or a mixture thereof may be used. Preferably a mixture in the range of 40-95% limestone is used. The lime and/or limestone is added in an amount such that the pH of the system is between about 7-12 and preferably about 8-10.

15 The present inventors have also surprisingly discovered that not only can the overall amount of leaching be increased but that such an increase can also be obtained by leaching under milder conditions than has hitherto been possible. Suitably the method of the  
20 present invention can be carried out at ambient pressure. This avoids the use of expensive pressure reactors and autoclave equipment. The preferred operating temperature is between about 50°C up to the boiling point of the mixture. Typically the maximum temperature is about  
25 95°C.

The leaching reaction can thus be carried out in open tank reactors. Excess heat is removed by evaporation of the solution. This avoids the need for costly heat exchangers. Heat can be easily introduced by  
30 known methods such as the injection of steam.

The leaching reaction is carried out in the presence of an oxygen containing gas. When the reaction is carried out in an open tank reactor the gas is typically introduced by sparging. The gas may be oxygen,  
35 air or oxygen enriched air. The gas flow is dependent upon the amount of oxygen required to sustain the leaching reaction and is typically about 0.01 - 0.5  $\mu$ m.

After the composition has been leached, the

mixture can be further treated by known methods to recover precious metals, principally by cyanide leaching.

Cyanidation occurs under alkaline conditions. As the leach solution is already alkaline, the solution  
 5 may be subjected directly to cyanide leaching. If desired the slurry may be thickened prior to cyanide leaching.

According to a second broad form of the invention there is provided a method of recovering  
 10 precious metals from a mineral composition comprising an iron containing refractory sulphide material, the method comprising;

grinding the material to a particle size  $P_{80}$  of at least 25 $\mu$ m;

15 leaching the ground material in the presence of lime and/or limestone and an oxygen containing gas; and  
 subjecting the leached material to a further leaching step to recover any precious metals.

#### BRIEF DESCRIPTION OF THE DRAWINGS

20 Figure 1 is a flow diagram of a preferred method of the present invention and Figure 2 is a flow diagram of a further preferred method of the present invention.

#### BEST MODE

25 The present invention will now be described in relation to the following examples. However, it will be appreciated that the generality of the invention as described above is not limited to the following examples.

30 **Example No. 1. Whole ore leach of a pyrite containing ore, with a 50:50 blend of limestone and lime, oxygen used as the oxidant.**

#### *Alkali Leach*

35 The ore sample was slurried in tap water at 50% solids and ground in a laboratory rod mill for a period of 20 minutes to achieve a slurry ground to 80% passing 106 microns. The slurry was then thickened to 60% solids,

and milled in a Netzsch horizontally stirred laboratory scale bead mill. The media used was 0.8 - 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 14  
5 microns. The final particle size distribution was determined by lasersizer.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the  
10 vessel. A 1,000 gram sample of the ground solids were added to the leach vessel along with 20 litres of tap water. The live volume of the reactor was 25 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry  
15 was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 2,000 cubic centimetres per minute.

20 The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from  
25 a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 35% w/w solids.

A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The  
30 controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was heated by steam injection into a  
35 jacket surrounding the vessel. The steam injection was regulated by a solenoid valve, which was controlled by an automatic temperature controller. The vessel temperature was controlled to 85°C. The outside of the vessel was

wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed.

- 5 A sample of the cake was analysed for remaining sulphide to determine the extent of pyrite oxidation. The sulphide analysis was determined both by evolution upon acidification, and by subtraction from total sulphur (by leco) and sulphate (by digest) The results of the test  
10 are listed in the table below. The dry weight of the filter cake was 1,150 grams.

Sample	Weight grams	% sulphide	% Pyrite	% sulphide oxidation
Feed	1000	8.2	15.2	90.1
Leach	1150	0.7	1.30	
Residue				

#### 15 *Cyanide Leach*

- A sample of the leach residue was slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton  
20 style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

- The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of  
25 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven and analysed to  
30 determine the precious metals recovery.

**Example No. 2. Leach of a pyrite containing concentrate, with a 50:50 blend of limestone and lime, oxygen used as**

the oxidant.

### *Alkali Leach*

The concentrate sample was slurried in tap  
5 water at 60% solids, and milled in a Netzsch horizontally  
stirred laboratory scale bead mill. The media used was  
0.8 - 1.1mm steel balls. Slurry was passed through the  
mill twice to achieve the required grind size of 80%  
passing 12 microns. The final particle size distribution  
10 was determined by lasersizer.

The leaching test was carried out in a  
cylindrical stainless steel reactor fitted with four  
baffles located equidistant around the edges of the  
vessel. A 200 gram sample of the ground solids were  
15 added to the leach vessel along with 2000mL of tap water.  
The live volume of the reactor was 2.5 litres. The  
vessel was fitted with a stainless steel lid to prevent  
evaporative loss from the reactor. The slurry was  
agitated by a 6 bladed Rushton style impeller. Oxygen  
20 was introduced to the reactor by an air spear, which  
terminated below the impeller. The oxygen flow was  
controlled by a rotameter at 400 cubic centimetres per  
minute.

The pH in the vessel was maintained by an  
25 automated pH analyser and controller, to a set point of  
10. A combination glass pH probe was immersed in the  
slurry through a port in the lid of the reaction vessel.  
A limestone/lime slurry was continuously circulated from  
a stirred beaker through a ring main using a peristaltic  
30 pump. The limestone/lime slurry consisted of 50% lime  
and 50% limestone, at a slurry density of 35% w/w solids.  
A solenoid valve was attached to the ring main, with an  
outlet line directed into the reaction vessel. The  
controller would actuate the solenoid when the pH in the  
35 vessel drifted away from the set point, and  
limestone/lime slurry was dosed into the vessel to return  
the pH to setpoint.

The vessel was placed on a thermocouple

controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel  
5 was wrapped in insulated foam to prevent heat loss. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was  
10 filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was 433 grams.

#### 15 *Cyanide Leach*

A sample of the leach residue was slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton  
20 style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of  
25 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven.

30

#### Results

Sample	% pyrite oxidised
Feed	NA
Oxidised Residue	90.1

The degree of sulphide oxidation was determined  
35 by XRD analysis. The precious metals extractions were determined by head and tails fire assay.

Mineral	Head	Residue
	% w/w	% w/w
quartz	9	5.3
pyrite	65	3.0
calcite		26.85
chlorite	3.4	4.9
gypsum	2.4	37.95
bassinite		1.55
muscovite	9	14.05
hematite		2.1
ankerite	3.6	
amorphous	2	6.5

Example No. 3. Leach of a pyrite containing concentrate,  
with a 50:50 blend of limestone and lime, air used as the  
5 oxidant.

#### Alkali Leach

The concentrate sample was slurried in tap  
water at 60% solids, and milled in a Netzsch horizontally  
10 stirred laboratory scale bead mill. The media used was  
0.8 - 1.1mm steel balls. Slurry was passed through the  
mill twice to achieve the required grind size of 80%  
passing 12 microns. The final particle size distribution  
was determined by lasersizer.

15 The leaching test was carried out in a  
cylindrical stainless steel reactor fitted with four  
baffles located equidistant around the edges of the  
vessel. A 200 gram sample of the ground solids were  
added to the leach vessel along with 2,000mL of tap  
20 water. The live volume of the reactor was 2.5 litres.  
The vessel was fitted with a stainless steel lid to  
prevent evaporative loss from the reactor. The slurry  
was agitated by a 6 bladed Rushton style impeller. Air  
was introduced to the reactor by an air spear, which  
25 terminated below the impeller. The air flow was



controlled by a rotameter at 600 cubic centimetres per minute.

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was 489 grams.

### *Cyanide Leach*

A sample of the leach residue was slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven.

10

### Results

Sample	% pyrite oxidised
Feed	NA
Oxidised Residue	90.22

The degree of sulphide oxidation was determined by XRD analysis. The precious metals extractions were determined by head and tails fire assay.

15

Mineral	Head	Residue
	% w/w	% w/w
quartz	9	5.8
pyrite	65	2.6
calcite		36.15
chlorite	3.4	3.8
gypsum	2.4	21.03
bassinite		14.65
muscovite	9	9.05
ankerite	3.6	
amorphous	2	6.9

Example No. 4. Leach of an arsenopyrite containing concentrate, with a 50:50 blend of limestone and lime, oxygen used as the oxidant.

20

#### Alkali Leach

The concentrate sample was slurried in tap water at 60% solids, and milled in a Netzsch horizontally

stirred laboratory scale bead mill. The media used was 0.8 - 1.1mm steel balls. Slurry was passed through the mill twice to achieve the required grind size of 80% passing 14 microns. The final particle size distribution was determined by lasersizer.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four baffles located equidistant around the edges of the vessel. A 1500 gram sample of the ground solids were added to the leach vessel along with 20L of tap water. The live volume of the reactor was 25 litres. The vessel was fitted with a stainless steel lid to prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 2000 cubic centimetres per minute.

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The limestone/lime slurry consisted of 50% lime and 50% limestone, at a slurry density of 30% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was heated by steam injection into a jacket surrounding the vessel. The steam injection was regulated by a solenoid valve which was controlled by an automatic temperature controller. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was 2965 grams.

### Cyanide Leach

A sample of the leach residue was slurried in tap water at a slurry density of 40% w/w solids in a baffled glass reactor. The volume of the reactor was 3 litres. The slurry was agitated by a 6 bladed Rushton style impeller. Air was introduced to the reactor by an air spear, which terminated below the impeller.

The pH of the slurry was adjusted and maintained at pH 10 using lime, and sodium cyanide was added to the slurry to maintain a free cyanide level of 500ppm. The test was carried out for a period of 24 hours. On completion of the test, the slurry was filtered and the filter cake washed with deionised water. The filtrate and washings were combined for analysis. The solid filter cake was dried in an oven.

### Results

Sample	% pyrite oxidised	% arsenopyrite oxidised
Feed	NA	NA
Oxidised Residue	90.6	>95

The degree of sulphide oxidation was determined by XRD analysis. The precious metals extractions were determined by head and tails fire assay.

Mineral	Head	Residue
	% w/w	% w/w
quartz	8.4	4.8
arsenopyrite	30.6	ND
pyrite	29.5	1.4

gypsum		38.4
bassinite		4.2
microcline	4.1	
jarosite		1.2
Scorodite		12.4
hematite		8.2
amorphous		28.7

Example No. 5. The Effects of Varying Alkali Mixtures on  
5 the Extent of Sulphide Oxidation

*Alkali Leach*

Approximately 5 kg of pyrite concentrate sample was  
slurried in tap water at 60 % solids, and milled in a  
10 Netzsch horizontally stirred laboratory scale bead mill.  
The media used was 0.8 - 1.1 mm steel balls. Slurry was  
passed through the mill twice to achieve the required  
grind size of 80% passing 8.6 microns. The final  
particle size distribution was determined by lasersizer.  
15 On completion of the grind, the ground slurry was split  
into 500 gram subsamples for the leaching test, and each  
subsample filtered separately. All subsamples were  
stored frozen to prevent oxidation of the pyrite.

A 200 gram sample was slit out of each  
20 subsample for the leaching testwork.

The leaching test was carried out in a  
cylindrical stainless steel reactor fitted with four  
baffles located equidistant around the edges of the  
vessel. A 200 gram sample of the ground solids were  
25 added to the leach vessel along with 2 litres of tap  
water. The live volume of the reactor was 2.5 litres.  
The vessel was fitted with a stainless steel lid to  
prevent evaporative loss from the reactor. The slurry  
was agitated by a 6 bladed Rushton style impeller. Oxygen  
30 was introduced to the reactor by an air spear, which

terminated below the impeller. The oxygen flow was controlled by a rotameter at 400 cubic centimetres per minute.

5 The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel. A limestone/lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic  
10 pump. The limestone/lime slurry composition was varied for the three leach tests according to the following ratios: 100% lime, 50% lime 50% limestone, 10% lime 90% limestone.

For all tests, the slurry density of the alkali  
15 blend was of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into  
20 the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by  
25 the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

30 On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was recorded.

35 The results are listed in Table 1, below

Table 1. The Effects Of Varying Lime/Limestone Blends On The Extent Of Pyrite Oxidation From Pyrite Flotation

## Concentrate

Alkaline Leach Tests				
	Test No.	Test 13	Test 20	Test 23
	pH	10	10	10
	Alkali mixture	100% lime	50% lime 50% limestone	10% lime 90% limestone
	Grind Size. 80% passing	8.6	8.6	8.6
	% solids	10.0	10.0	10.0
Feed data:				
	Dry solids wt g	200	201.0	201.1
	% pyrite in head	65.0	65.0	65.0
Residue data:				
	% pyrite in residue	3.2	3.0	1.0
	% pyrite oxidised from XRD	95.47	90.1	94.3

Example No. 6. The Effects of Varying Grind Size on the  
5 Extent of Sulphide Oxidation.

*Alkali Leach*

Approximately 4 kg of pyrite concentrate sample was  
slurried in tap water at 60% solids, and milled in a  
10 Netzsch horizontally stirred laboratory scale bead mill.  
The media used was 0.8 - 1.1 mm steel balls. Slurry was  
passed through the mill in five consecutive passes, with  
a subsample taken from the ground slurry from each pass.  
Each subsample therefore was ground to a different  
15 particle size. The particle sizes from each pass were:

Grind Size -	51.9	32.04	17.94	13.64	11.71	8.6
80% passing						

The final particle size distribution was

determined by lasersizer. On completion of the grind, the ground slurry was split into 500 gram subsamples for the leaching test, and each subsample filtered separately. All subsamples were stored frozen to prevent  
5 oxidation of the pyrite.

A 200 gram sample was slit out of each subsample for the leaching testwork.

The leaching test was carried out in a cylindrical stainless steel reactor fitted with four  
10 baffles located equidistant around the edges of the vessel. A 200 gram sample of the ground solids were added to the leach vessel along with 2 litres of tap water. The live volume of the reactor was 2.5 litres. The vessel was fitted with a stainless steel lid to  
15 prevent evaporative loss from the reactor. The slurry was agitated by a 6 bladed Rushton style impeller. Oxygen was introduced to the reactor by an air spear, which terminated below the impeller. The oxygen flow was controlled by a rotameter at 400 cubic centimetres per  
20 minute.

The pH in the vessel was maintained by an automated pH analyser and controller, to a set point of 10. A combination glass pH probe was immersed in the slurry through a port in the lid of the reaction vessel.  
25 A lime slurry was continuously circulated from a stirred beaker through a ring main using a peristaltic pump. The lime slurry was of 35% w/w solids. A solenoid valve was attached to the ring main, with an outlet line directed into the reaction vessel. The controller would actuate  
30 the solenoid when the pH in the vessel drifted away from the set point, and limestone/lime slurry was dosed into the vessel to return the pH to setpoint.

The vessel was placed on a thermocouple controlled hotplate, with the thermocouple immersed in  
35 the slurry through a stainless steel sheath. The vessel temperature was maintained at the required temperature by the thermocouple controller. The outside of the vessel was wrapped in insulated foam to prevent heat loss. The



vessel temperature was controlled to 85°C. The outside of the vessel was wrapped in insulated foam to prevent heat loss.

On completion of the test, the slurry was filtered and the filter cake dried and weighed. A sample of the cake was sent for XRD analysis to determine the extent of pyrite leaching. The dry weight of the filter cake was recorded.

The results are listed in Table 2, below

Table 2. The Effects of Varying Grind Size on the Extent of Pyrite Oxidation from Pyrite Flotation Concentrate

Alkaline Leach Tests						
Test No.	10	24	11	12	15	13
pH	10	10	10	10	10	10
Alkali mixture	100% lime	100% lime	100% lime	100% lime	100% lime	100% lime
Grind Size . 80% passing	51.9	32.04	17.94	13.64	11.71	8.6
% solids	10.0	10.0	10.0	10.0	10	10.0
Feed data:						
Dry solids wt g	200.0	200	200	200	200	200
% pyrite in head	65.0	65.0	65.0	65.0	65.0	65.0
Residue data:						
% pyrite in residue	16.7	9.7	7	2.7	3.7	3.2
% pyrite oxidised from XRD	58.9	80.1	91.06	96.81	95.54	95.47

It can be seen that by grinding 25µm or less 90% or more of the pyrite is oxidised. A high level of pyrite oxidation typically translates to high precious metal recovery.

The methods of the present invention offer a number of advantages over existing methods. First, iron containing refractory ores such as pyrite can be leached

to high levels under alkaline conditions using lime and/or limestone as the alkali source. Lime and limestone offer significant economic advantages over the known agents such as caustic. For example, current costs  
5 for caustic are about AUD\$440 per tonne, ammonium based salts, about AUD\$450, lime AUD\$100-200 and limestone AUD\$15-41/tonne.

Further, the leach need not be pressurised which translates to significant capital and operating  
10 costs. Still further, costs can be reduced as the leach can be carried out using air instead of oxygen. This means that oxygen need not be purchased or produced.

The leach residence times for the preferred methods of the present invention are typically about a  
15 few hours.

These residence times compare favourably to oxidative leaching under acidic conditions and are in fact superior to atmospheric ferric leaching.

During the leaching iron precipitates as  
20 goethite and haematite, rather than jarosite as occurs under acidic conditions. Jarosite inhibits subsequent precious metal recovery by making the residue difficult to settle and filter. Further, jarosite is an environmentally acceptable residue.

Gypsum is also formed during the leach and  
25 precipitates. An advantage of gypsum is that it enhances the filterability of the residue. Gypsum is not formed when using conventional alkali reagents.

Another advantage over the use of the  
30 conventional alkali reagents is that in the present invention arsenic present in the refractory material is precipitated as calcium arsenate. The level of arsenic in the leach liquor is typically below detectable limits. When using conventional alkalis arsenic is present in the  
35 leach liquor.

It should be appreciated that various other changes and modifications may be made to the embodiment described without departing from the spirit and scope of

the invention as described.

DATED this Twenty First day of September 1998

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M.I.M. HOLDINGS LIMITED

By their Patent Attorneys  
CULLEN & CO.

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